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Fabrication of Nano-meter-sized Periodic Structures by Holographic Exposure of Pulsed Laser Light and Their Novel Optical properties and Lasing

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By means of one-shot holographic exposure of pulsed laser beams, permanent and transient nano-meter-sized periodic structures were fabricated, and studies on the application for various optical devices were curried out. Using nega-type photoresist SU-8 as a sample, one- and two-dimensional periodic structures with near vertical sidewalls and high aspect ratio were fabricated. Moreover, laser action utilizing transient grating was studied. The laser action was realized by a holographic excitation under a first-order diffraction condition, using a film of conducting polymer MDDO-PPV as an active medium. Lasing wavelength can be tuned by changing the angle between two excitation laser beams. A permanent periodic structure can also be written by intense exposure. An electro-tuning of lasing wavelength under holographic excitation was also realized utilizing a dye-doped nematic liquid crystal as an active medium. 30 nm shift of lasing wavelength could be achieved with an applied voltage of several volts.

KEYWORDS: photonic crystal; holography; conducting polymer; SU-8; liquid crystal; DFB laser; holographic excitation

パルス干渉露光によるナノ周期構造の作製とその光学的性質ならびにレーザー発振

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パルス干渉露光により、定常的あるいは過渡的なナノ周期構造の作製を試み、その様々な光デバイスへの応用の検討を行った。試料としてネガ型フォトレジスト SU-8を用い、良好な垂直プロファイル、高いアスペクト比を有する一次元・二次元周期構造の作製に成功した。さらに、過渡的な干渉場を利用したレーザー発振についても検討を行った。レーザー活性媒質として、導電性高分子 MDDO-PPV 薄膜を用い、一次の回折条件下における干渉励起によるレーザー発振の観測に成功した。励起二光束間角度により、発振波長の制御が可能である。また、強励起により定常的な一次元周期構造の書き込みも可能である。さらに、レーザー色素をドープしたネマティック液晶をレーザー活性媒質として用い、干渉励起下におけるレーザー発振波長の電界制御にも成功した。数ボルト程度の低電圧により、およそ30nm もの発振波長のシフトが実現可能である。

キーワード：フォトニック結晶；ホログラフィー；導電性高分子；SU-8；液晶；DFB レーザー；干渉励起

1. Introduction

Since proposals of Yablonovitch[1] and John[2], a photonic crystal (PC), which is made from a periodic dielectric structure with a periodicity in a range of optical wavelength, has attracted much attention from both fundamental and practical points of view[3] (Fig. 1 (a)). In the PC, the propagation of light is inhibited under the Bragg condition, which results in an appearance of an optical stop band or a photonic band gap (PBG). In the PBG, spontaneous emission is inhibited, so that a low-threshold laser action can be expected.[1,4]

For the fabrication of these PCs, micro - fabrication techniques of semiconductors have been widely employed so far.[5-7] Utilizing semiconductor such as Si, GaAs, high contrast of refractive index of composing material, which is key-point for the control of properties of PCs, can be achieved. However, these techniques require complicated procedures and multi-steps for the fabrication.

Recently, holographic technique has attracted much attention for the easy fabrication technique of PCs.[8-10] Not only the ease of fabrication, this technique possesses many advantages to the other techniques as follows. Various structures can be realized by changing the parameters of interfering laser lights, such as the number of laser beams, intensity, wave vector (direction), polarization, phase, and so on (Fig. 1 (b)). More over, filling factor can also be controlled.

In this paper, by means of one - shot holographic exposure of pulsed laser beams, various nano-meter-sized periodic structures were fabricated, and studies on the application for PCs and various optical devices were curried out. In Sec. 2, experimental procedures of this study are summarized, and in Sec. 3, experimental results are shown. In 3.1, fabrication of one- and two-dimensional periodic structures with near vertical sidewalls and high aspect ratio were demonstrated using nega-type photoresist SU-8 as a sample. In 3.2, laser action utilizing transient grating was studied. The laser action was realized by a holographic excitation under a first-order diffraction condition, using a film of conducting polymer MDDO-PPV as an active medium. Tuning of lasing wavelength by changing the angle between two excitation laser beams is also demonstrated. In 3.3, an electro-tuning of lasing wavelength under holographic excitation was demonstrated utilizing a dye-doped nematic liquid crystal as an active medium. Finally, in Sec. 4, summaries of our studies are given.

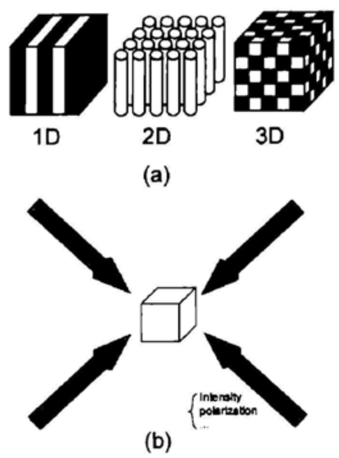


Fig.1: (a) Schematic representation of photonic crystal. (b) Schematic representation of fabrication of photonic crystal by 4-beam interference

図1. (a) フォトニック結晶 (b) 4光束干渉露光によるフォトニック結晶の作製の概念図

2. Experiment

2.1 Samples and device structure

For the fabrication of one- and two-dimensional periodic structures, we used an epoxy based nega-type photoresist, SU-8 2005 (Micro Chem),[11] which has been widely used in the field of MEMS (micro electrical mechanical system) so far. SU-8 has very high optical transparency above 360 nm, so that high aspect ratio imaging with near vertical sidewalls can be achieved easily even by one-photon reaction.

Fabrication processes are summerized as follows:

(1) Spin-coating of sample on glass substrate
(2) Soft bake (65°C and 95°C)
(3) Exposure
(4) Post exposure bake: PEB (65°C and 95°C)
(5) Develop(using diacetonealcohol as a developer)
(6) Rinse and dry

Upon exposure, cross-linking proceeds in two steps, (1) formation of a strong acid during the exposure process, followed by (2) acid-initiated, thermally driven epoxy cross-linking during the PEB step.

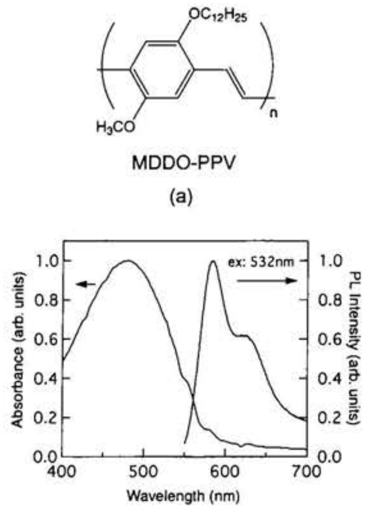


Fig.2: (a) molecular structure (b) absorbance and photoluminescence spectra of conducting polymer MDDO-PPV used in this study.
図２．実験に用いた導電性高分子 MDDO-PPV の(a) 分子構造 (b) 吸収蛍光スペクトル

In Fig. 2 (a), the molecular structure of conducting polymer, MDDO-PPV, used for the transient lasing experiment described in 3.2, is shown. Spectra of absorbance and photo luminescence (PL) of MDDO-PPV are also shown in Fig. 2 (b). The sample was dissolved in solvents such as *p*-xylene and spin-coated on the quartz glass substrates.

For the demonstration of the electric tunability of the lasing, the NLC mixture E-44 (Merck), which shows a nematic phase in room temperature, was used as a host material. The dielectric anisotropy of this NLC is positive so that if an electric field is applied to LC layer, LC molecules align their molecular long axis along the applied electric field. In Fig. 3, a molecular structure of a laser dye dopant, [2-[2-4-(Dimethylamino)phenyl]ethenyl]-6-methyl-4H-pyran-4-ylidene] propanedinitrile, DCM (Exciton) was shown. The concentration of the dye was 0.7 wt.-%. The ordinary and extraordinary refractive indices n_o and n_e of this mixture are 1.53 and 1.78, respectively (at 632.8 nm). The sample was inserted in the isotropic phase by a capillary

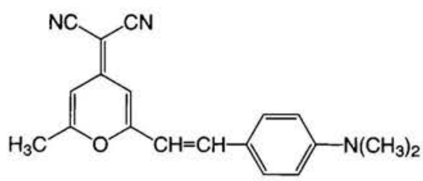


Fig.3: molecular structure of laser dye DCM used in this study.
図３．実験に用いたレーザー色素 DCM の分子構造

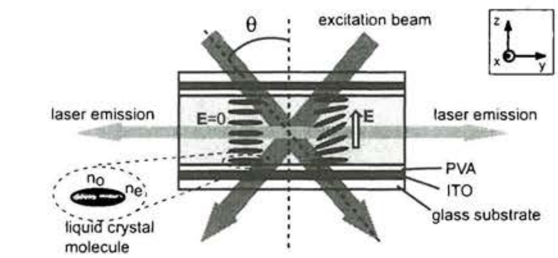


Fig.4: Schematic representation of the cell structure used in this study for the electrical tuning of the laser emission upon holographic excitation.
図４.干渉励起下レーザー発振の電界制御に用いたセルの構成図

action into the sandwiched cell that is composed of two Indium-Tin-oxide (ITO)-coated quartz glass substrates. The cell structure is shown in Fig. 4. The cell gap was 2.5 μm. In order to obtain a planarly aligned cell in which the direction of LC initially aligns parallel to the substrates, the surfaces of substrates were coated with a poly(vinyl alcohol) (PVA) and rubbed. The PVA layer also acted as a low index clad layer (n_{PVA} = 1.52). In such a cell structure, the LC layer acts as a core layer of the slab waveguide and the electromagnetic (EM) wave can be confined effectively in the LC layer under the total reflection condition. [12,13]

2.2 One-shot holographic exposure system for the fabrication of periodic structure

For the one-shot holographic exposure, a third harmonic light of Q-switched Nd:YAG laser (Spectra Physics, Quanta-Ray INDI) was used as a light source, whose wavelength, pulse width and pulse repetition frequency were 355 nm, 8 ns and 10Hz, respectively. Only one pulse was exposed to the sample. The polarization of both two excitation laser beams was set to be s-polarization, that is, perpendicular to the incident plane, in order to make photoinduced transient grating to be pure intensity grating.[14,15] The beam was expanded using a pair of convex and concave lenses and passed a small aperture in order to use only middle part of Gaussian beam, so that homogeneous illumination can be realized. The Al mirror was placed in front of the sample cell perpendicular to the cell, so that so-called Lloyd's mirror setup for the holographic illumination was realized. The angle between two writing laser beams was kept to be 20°, so that the pitch of the formed periodic structure was about 1 μm. This setup is similar to that of holographic excitation, so detailed description will be given in next section.

2.3 Experimental setup of optical measurement of Lasing

Figure 5 shows the experimental setup for emission measurement. A third harmonic light of Q-switched Nd:YAG laser (Spectra Physics, Quanta-Ray INDI) was used for an excitation of MDDO-PPV and dye-doped NLC, whose wavelength, pulse width and pulse repetition frequency were 355 nm, 8 ns and 10Hz, respectively. The polarization of both two excitation laser beams was set to be s-polarization as described above, in order to make photoinduced transient grating to be pure intensity grating. The excitation laser beams were focused into a stripe using a cylindrical lens. The Al mirror was placed in front of the sample cell perpendicular to the cell, so that so-called Lloyd mirror setup for the holographic illumination was realized as shown in the inset of Fig. 5. The sample (MDDO-PPV spin-coated film or dye-doped NLC cell) and mirror were fixed on a rotational θ stage, and the angle between two excitation laser beams 2θ was adjusted by the rotational angle of this stage.

The emission spectra from the sample were measured utilizing a spectrograph with a charge-coupled device (CCD) detector (Oriel, Multi Spec 257) having a spectral resolution of 0.3 nm. For the application of an electric field to the dye-doped NLC

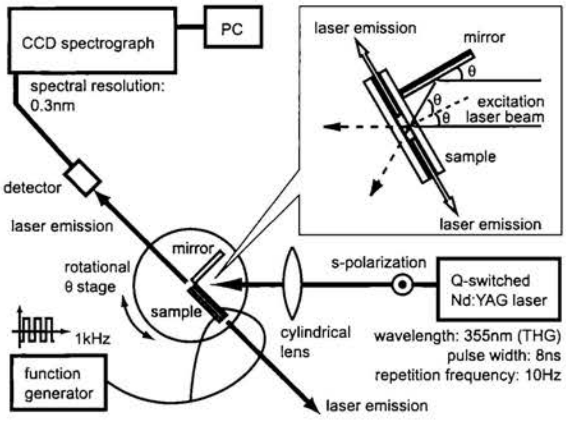


Fig.5: Schematic representation of the experimental setup for emission measurement. Inset: the Lloyd's mirror setup for the interferential illumination.
図5. 発光特性測定系,挿入図：ロイドの鏡干渉露光系

sample cell, a function generator (Hewlett Packard, 3314A) was used. A rectangular shaped AC voltage of 1 kHz was applied.

3. Results and Discussion

3.1 Fabrication of one- and two-dimensional periodic structure

In this section, the results of the fabrication of one- and two-dimensional periodic structures using nega-type photoresist SU-8 as a sample are shown.

Figure 6 shows the SEM images of formed one-dimensional periodic structures. Parameters for the fabrication (Fig.6 (a)-(d)) were as follows: exposure, 8.9 mJ/cm^2; PEB, 90 sec at 65°C, 90 sec at 95°C; develop, 60 sec in ultrasonic bath. It can be seen that high aspect ratio imaging with near vertical sidewalls was achieved.

It was found that the quality of formed periodic structures strongly depend on parameters such as exposure dose, baking time, and so on. Especially, PEB time is quite important. Even if exposure was performed equally, filling factor of formed structure differed when PEB time was different. As mentioned in Sec. 2, upon exposure cross-linking proceeds in two steps as follows. (1) formation of a strong acid during the exposure process, (2) acid-initiated, thermally driven epoxy cross-linking during the PEB step. In the PEB process, diffusion of photo-acid might be occur, so PEB time affect the filling factor of formed structure. Further

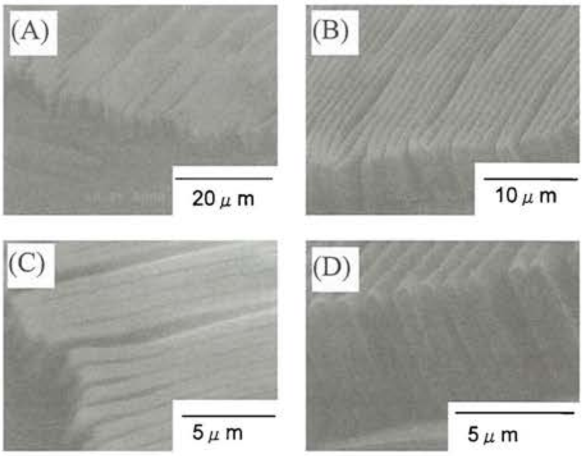


Fig.6: SEM images of formed one-dimensional periodic structures on the nega-type photoresist SU-8.
図6.ネガ型フォトレジスト SU-8上に作製した1次元周期構造の SEM 像

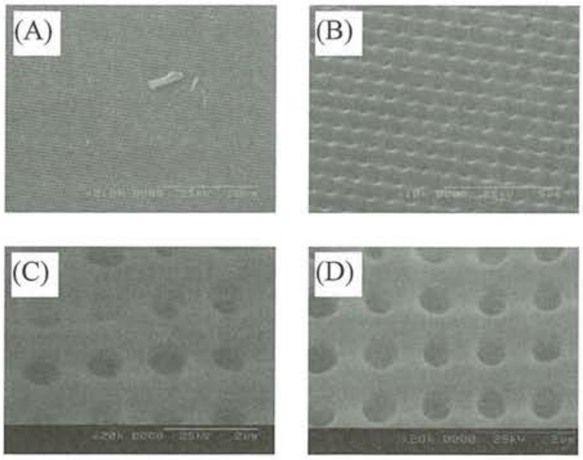


Fig.7: SEM images of formed two-dimensional periodic structures on the nega-type photoresist SU-8.
図７.ネガ型フォトレジスト SU-8上に作製した２次元周期構造の SEM 像

optimization of fabrication parameters is needed.

Figure 7 shows the SEM images of formed two-dimensional periodic structures. Parameters for the fabrication were as follows: exposure, 7.6 mJ/cm^2; softbake, 3 min at 65°C, 5 min at 95°C; PEB, 5 min at 65°C, 15 min at 95°C; develop, 60 sec in ultrasonic bath and stirrer bath (Fig. 7 (a), (b)) and exposure, 7.6 mJ/cm^2; softbake, 3 min at 65°C, 5 min at 95°C; PEB, 1 min at 65°C, 15 min at 95°C; develop, 10 min in stirrer bath (Fig. 7 (c), (d)). From Fig. 7 (a) and (b), it can be seen that homogeneous structures were obtained in wide area. Figures 7 (c) and (d) also show that holes of round shape with near vertical sidewalls were obtained. These structures can be applied to the two-dimensional PCs.

3.2 Laser action of conducting polymer film by holographic excitation

In 1971, Kogelnik *et al.* demonstrated the laser action upon photoexcitation of rhodamine 6G doped gelatin film with holographically inscribed phase grating.[16] In their device, the optical feedback was provided by backward Bragg scattering from phase grating and mirrorless distributed feedback (DFB) laser action was observed. They also showed the DFB laser action could be achieved by a transient grating using interference fringes induced by two excitation laser beams (holographic excitation).[17] The wavelength of laser emission λ_{Bragg} upon holographic excitation can be expressed by the following equation,

$$\lambda_{Bragg} = n_{eff}\lambda_{ex}/m\sin\theta, \qquad (1)$$

where n_{eff} is the effective refractive index of active layer, λ_{ex} is the wavelength of excitation beams, m is the order of diffraction and θ is the half angle between two excitation beams. Based on this equation, the tunability of laser emission wavelength was also demonstrated by changing the angle between two excitation beams 2θ or refractive index of laser dye solution n_{eff}. In this section, lasing from a thin film of MDDO-PPV by holographic excitation was studied.[18]

For the excitation of the sample, a Q-switched Nd:YAG laser was used as described above. When THG (355 nm) and SHG (532 nm) light are used for the excitation light source, lasing wavelength under first ($m=1$) and second ($m=2$) diffraction conditions can be estimated using equation (1) as shown in Fig. 8 (a) and (b), respectively. The dark area in these figures shows the tunable range of lasing wavelength

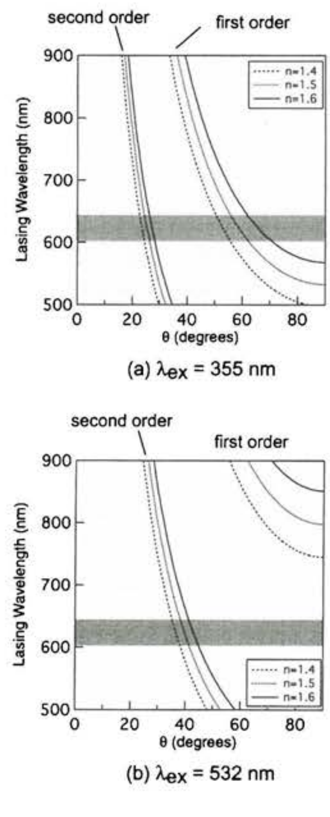


Fig.8: Theoretical estimation of lasing wavelength under holographic excitation under first and second diffraction conditions when wavelength of excitation laser light are (a) 355 nm (b) 532 nm.
図８．励起波長 (a) 355 nm (b) 532 nm の場合の干渉励起によるレーザー発振波長の理論値

from MDDO-PPV. When THG light is used as an excitation light source, both first and second order diffraction condition can be satisfied as shown in Fig. 8 (a). On the other hand, when SHG light is used, only second order diffraction condition can be realized as shown in Fig. 8 (b). We have carried out the experimental study using both THG and SHG light as a light source, however, laser action was observed only under first order diffraction condition by using THG light. This shows that the first order diffraction condition is essential for the realization of lasing.

Figure 9 shows the emission spectra from MDDO-PPV film at various angles between two excitation laser beams. The sharp peaks were observed in the broad PL spectra and that peak showed blue shift when the angle between two excitation laser beams was expanded. The peak wavelength agreed with theoretical estimated ones under first order diffraction condition, so that this clearly shows that the laser action was realized by holographic excitation. The tunable range of lasing wavelength was about 30 nm. On the other hand,

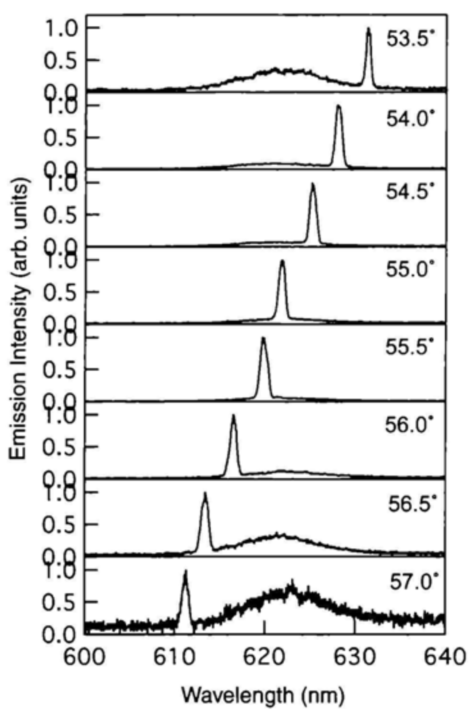


Fig.9: Emission spectra from MDDO-PPV film at various angles between two excitation laser beams.
図９．様々な励起二光束間角度における発光スペクトル

we have reported previously, in the study on the DFB lasing from MDDO-PPV on the photoinduced surface relief grating (SRG) structure on photochromic azopolymer film, that lasing was observed only when the diffraction condition agreed with around the 0-1 vibration mode of MDDO-PPV.[19] In that case, the height of SRG was about 100 nm. This might be too high for the DFB lasing.

When excitation of sample was performed intensely, permanent grating structure could also be recorded. However, whether this grating structure were refractive grating or surface relief grating is not clear yet. In order to clarify the formation mechanism of this permanent grating, further studies are required such as observation of the surface of the film by AFM and so on.

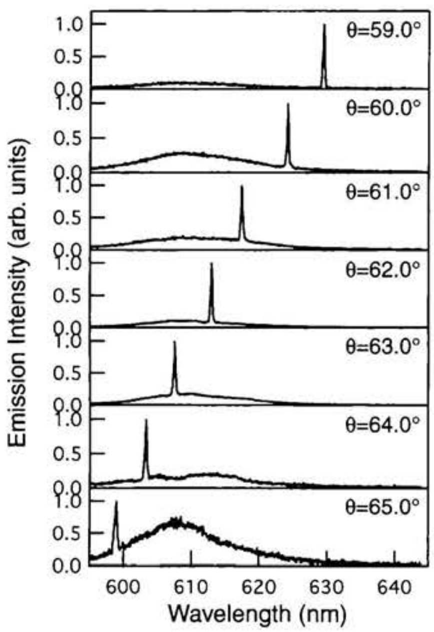
3.3 Tunable laser action of nematic liquid crystal doped with laser dye by holographic excitation

From the equation (1), it can also be expected that, if the effective refractive index of the active medium n_{eff} is electrically changed, electrical tuning of laser emission upon holographic excitation can be realized. In this section, we propose an electrical tuning of laser emission using a dye-doped nematic liquid crystal (NLC) as an active medium as schematically shown in Fig. 4.[20-22]

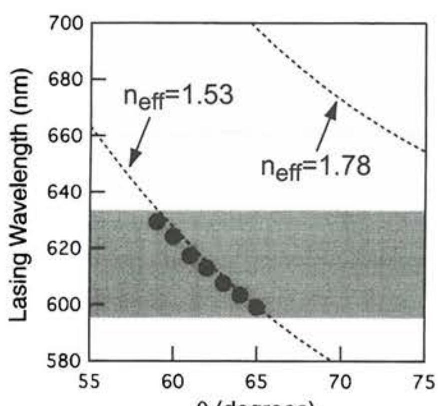
Liquid crystal (LC) molecules have elongated rod-like shape, which result in an appearance of an optical anisotropy, that is, LC molecules have an extraordinary refractive index n_e along the molec s ular long axis (director) and an ordinary refractive index n_o perpendicular to the director as shown in the inset of Fig. 4. LC also has anisotropy in dielectric constant so that the director of LC molecules can be controlled by the applied electric field.[23] This indicates that, if LC is used as an active material, the effective refractive index of the laser medium n_{eff} can be electrically controlled due to the field-induced reorientation of LC molecules.

In the configuration shown in Fig. 4, the rubbing direction in the NLC cell was perpendicular to the rotational axis of the stage. That is, the director of NLC molecules in the cell lies in the incident plane of two excitation beams. Under such an illumination condition, both transverse electric (TE) and transverse magnetic (TM) -guided modes in the NLC core layer feels the ordinary refractive index n_o of the NLC in the absence of the electric field.

Figure 10 (a) shows the emission spectra of the dye-doped NLC waveguide for various excitation angles (from 59.0° to 65.0° by 1.0°) above the threshold pump pulse energy without applied voltage. The full width at half maximum (FWHM) of the emission peak is about 0.5 nm, which is limited by the spectral resolution of our experimental setup. It was shown that the tunability of lasing wavelength upon holographic excitation was about 30 nm. In Fig. 10 (b), the excitation angle dependence of the lasing wavelength is also shown. The shaded region shows the tunable range of this laser. The dashed lines show the theoretical curves of the lasing wavelength under the first order diffraction condition ($m=1$) assuming the effective refractive indices n_{eff} as 1.53 and 1.78. The excitation angle dependence of the lasing wavelength agrees well with the theoretical curve with the effective refractive index n_{eff} of 1.53. It was confirmed that the laser emission wavelength could be tuned by changing the excitation angle and that the DFB laser action was realized due to the holographically induced transient grating.



(a)



(b)

Fig.10: (a) Emission spectra of the dye-doped NLC waveguide upon holographic excitation at various excitation angles without applied voltage. (b) the excitation angle dependence of the lasing wavelength.

図10. (a) 様々な励起二光束間角度における色素ドープしたネマティック液晶導波路の電界無印加時における発光スペクトル (b) レーザー発振波長の励起二光束間角度依存性

Figure 11 shows the emission spectra of the dye-doped NLC waveguide at various applied voltages. The half angle between two excitation laser beams θ was fixed to be 65.0°. When the applied voltage was below 0.7 V, laser emission peak was observed at 599 nm (peak 1) and no change was observed in the laser emission. When the applied voltage exceeded 0.8 V, another new peak (peak 2) appeared in longer wavelength. The peak 2 showed a continuous red-shift with increasing the applied voltage. Above the applied voltage of about 1.0 V, laser emission of peak 2 became multi-mode. When applied voltage was more than about 1.4 V, laser emission disappeared. On the other hand, the peak 1 did not shift at all. The amplitude of peak 1 decreased gradually, and laser emission peak 1 finally disappeared above 1.1 V. Applied voltage dependence of the lasing wavelength of peak 1 (open circle) and peak 2 (closed circle) is summarized in Fig. 12. The threshold voltage for the appearance of peak 2 is observed at about 0.8 V, which should be attributed to the Frederiks transition of the NLC. The electrical tuning of laser action could be performed reversibly.

In order to explain these results, TE- and TM-guided mode should be treated separately. The

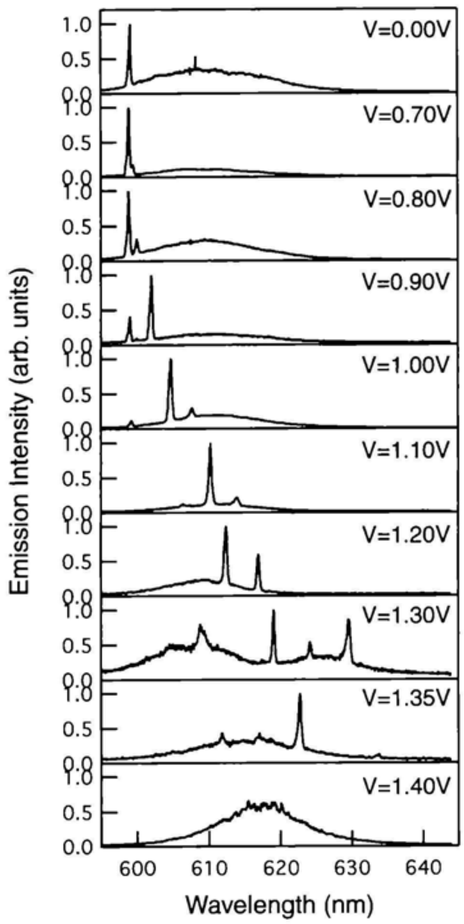


Fig.11: Emission spectra of the dye-doped NLC waveguide upon holographic excitation at various applied voltages (θ= 65.0°)
図11. 様々な印加電圧における色素ドープしたネマティック液晶導波路の発光スペクトル (θ= 65.0°)

reorientation of NLC molecules upon the application of an electric field takes place in the *xz*-plane, so that the effective refractive index n_{eff} for TM-guided mode increases gradually with the field-induced reorientation of the NLC, the lasing wavelength associated with TM-guided mode should shift to longer wavelength. Consequently, the red-shift of the lasing peak 2 might be attributed to TM-guided mode. With the increase of the effective refractive index n_{eff}, the effective thickness of the core layer also increases and higher order guided mode should appear. The appearance of multi-mode emission in peak 2 upon application of more than about 1.0 V can be explained in terms of this higher order guided mode. At more than 1.4 V, the diffraction condition for the DFB laser action could not be satisfied in the tunable range of this laser anymore, so that the laser action was not

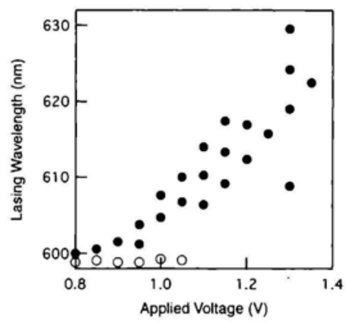


Fig.12: The applied voltage dependence of lasing wavelength.
図12. レーザー発振波長の印加電圧依存性

observed.

On the other hand, TE-guided mode feels the ordinary refractive index n_o of the NLC regardless of the applied voltage, so that no change in lasing wavelength of peak 1 should be attributed to TE-guided mode. Although no change of lasing wavelength was observed, the lasing intensity of peak 1 was reduced and peak 1 disappeared at more than about 1.1 V. This is attributed to the red-shift of the photoluminescence (PL) spectrum of the doped laser dye caused by the increase in the effective refractive index of the NLC core, which can also be seen in Fig 11. Due to the shift of the PL spectrum, attenuation of optical gain was caused. In order to clarify them, numerical analyses based on waveguide mode theory were performed. The results of them is to be published elsewhere.[21] Moreover, single-mode operation of this laser was also realized using low index NLC sample.[22]

Using this holographic excitation technique, fabrication of sub-micrometer sized periodic structure for the DFB laser action is not nec s essary, and the bulky fluidic characteristic of LC can be effectively utilized. By varying cell configurations such as cell gap, initial alignment of LC, switching procedure of LC and so on, various lasing modes based on the waveguiding mode theory can be realized and controlled.

4. Conclusions

In conclusion, by means of one-shot holographic exposure of pulsed laser beams, permanent and transient nano-meter-sized periodic structures were fabricated, and studies on the application for various optical devices were curried out.

Using nega-type photoresist SU-8 as a sample, one- and two-dimensional periodic structures with near vertical sidewalls and high aspect ratio were fabricated. These structures can be applied one- and two-dimensional PCs.

A laser action utilizing transient grating was studied. The laser action was realized by a holographic excitation under a first-order diffraction condition, using a film of conducting polymer MDDO-PPV as an active medium. Lasing wavelength can be tuned by changing the angle between two excitation laser beams. A permanent periodic structure can also be written by intense exposure. The difference between DFB lasing based on SRG structure was also discussed.

An electro-tuning of lasing wavelength under holographic excitation was also realized utilizing a dye-doped NLC as an active medium. Upon an applied electric field, the wavelength of the laser emission was continuously tuned on the basis of the reorientation of NLC molecules. 30 nm shift of lasing wavelength could be achieved with an applied voltage of several volts.

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